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A FEASIBILITY STUDY ON BIODIESEL PRODUCTION FROM WASTE BEEF SUBCUTANEOUS FATS

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ABSTRACT

This paper deals with the feasibility study of biodiesel production from the tallow rendered from the subcutaneous wastes. The subcutaneous wastes were collected from leather tanneries and animal slaughter houses. The fat content was determined based on the proximate analysis of the collected wastes and various rendering techniques were employed to study the rendering effectiveness. Base catalysed single step transesterification was carried out for biodiesel production. The characterization and quantification of biodiesel was done with the help of FT-IR and GC-MS. Maximum fat content in subcutaneous wastes was found to be 92.84%. The dry rendering technique was found to be very effective than compared to the wet rendering technique and was found to be 18.04% greater than wet rendering technique. The most optimised transesterification parameters were found to be (i) molar ratio: 9.5, (ii) catalyst concentration: 3%, (iii) reaction temperature: 65^{O}C and (iv) reaction time: 70 minutes. The confirmation of biodiesel was concluded from the high intensity peaks in the FT-IR spectra at 1740cm^{-1} and 1196cm^{-1} which corresponds to the C=O stretching and O-CH_3 group. Ethyl Oleate, Ethyl Palmitate, Ethyl Stearate and Ethyl Myristate were identified as the dominant fatty acids with availability of 40.25%, 21.26%, 17.41% and 3.59% respectively. Based on the results, it can be concluded that possibility of producing biodiesel from subcutaneous wastes was high and can be a viable replacement as low cost feedstock from waste.

KEYWORDS: Subcutaneous Wastes, Leather Fleshing, Beef Tallow, Ethanol Based Transesterification & Fatty Acid Esters

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Original Article

INTRODUCTION

Development of alternate fuels have satisfied the problems related to energy demand to meet the global need and increasing depletion of fossil fuels. Biodiesel is one such alternate fuel with robustness and high sustainability. This biofuel is highly bio-degradable, non-toxic with enhanced engine performance along with reduced emissions and engine parts wear, which makes it more desirable for combustion based applications [1].

These superior qualities of biodiesel have gained attention throughout the world and numerous researches related to its production[2], optimization [3], detailed study on Performance, combustion &the emission characteristics of engine [4,5], unconventional applications[6,7] have been carried out to commercialize this renewable fuel at a minimised retail price. However, reducing the overall cost is a challenging task and can be reduced by either employing low cost feedstocks or introducing efficient production techniques. Selection of low cost feedstocks is regarded as the sound option and must be chosen without compromising the "food vs. fuel "conflict. Waste edible seed and plant oils [8-10], waste animal fats[11-14], residual oils, waxes and greases [15-17] are the most promising low cost feedstocks with good lipid content in it. Among these, waste animal fats are the most successful low cost feedstock owing to its easy and abundant availability from various sources like animal slaughter houses, meat processing industries [18] and leather tanning units[19].

Specifically, waste beef fat is regarded as high potential feedstock available in abundance and can be used for large scale biodiesel production. In general, these waste beef fat are rendered from the wastes discarded from tanneries, animal slaughter houses and meat processing units; and are pre-treated, refined before being subjected for transesterification reaction. On average, 60,000 tonnes of solid wastes, mostly from beef & cattle, are generated from leather tanneries, in which 40-50% of these wastes compromises of fleashing wastes, rich in fat content [20]. Similarly, in case of animal slaughtering for current year, 2.45 Million metric tonnes of beef and cara beef were slaughtered for food consumption purposes throughout the country out of which 1.85 Million metric tonnes were exported to the countries who serves as global beef customers [21].

Production of biodiesel from waste beef tallow is done using transesterification reaction, which is carried out in different ways for improvising the overall yield. For instance, Fröhlich et al., 2010 carried out single stage base catalysed transesterification on beef tallow with FFA content of 11% using methanol as solvent [22]; In contrast to that, Bhatti et al., 2008 carried out acid catalysed transesterification on beef tallow, where 2.5g of concentrated sulphuric acid was used as catalyst for a reaction time of 6 hours [23]. In addition, two stage base catalysed transesterification was carried out on beef tallow using methanol as solvent [24]. Arajuo et al., produced biodiesel from 100g of beef tallow by initially micro-emulsifying with 10ml of methanol followed by addition of potassium methoxide (15ml/2.5 g) and refluxing for 45 minutes. Apart from the production process, the biodiesel was characterised for various fatty acid esters present in the tallow and their thermo-physical properties were evaluated [25]. Similarly, the biodiesel production, characterization of fatty acid esters and fuel properties were determined for beef tallow produced by acid base catalysed transesterification using methanol as solvent to study the influence of acid catalyst on biodiesel properties[26]. Post analysis, the biodiesel produced from waste beef tallow was tested for determining its performance, combustion and emission characteristics and was found to be very efficient for engine application. The most optimised blend for engine application for both commercialization and testing purpose was found to be B20 (80% diesel + 20% biodiesel).[5,27-28]. Ultimately, the use of beef tallow has reduced the threats to the environment and has found to be having energy, environmental and economic advantages.

In this present study, the feasibility of biodiesel production from subcutaneous wastes was studied and the reaction parameters were optimised by means of response surface method. A novel approach of combining fats from two different sources have been tried for biodiesel production and this could provide a clear description about the multiple source feedstocks for biodiesel production.

MATERIALS AND METHODOLOGY

Collection of Sample

The discarded subcutaneous wastes were collected from the leather tanneries and animal slaughter houses in and around Vellore, India. The collected wastes were carefully preserved under refrigeration to prevent rancdification of fats and avoid pathogenic infections.

Proximate Analysis of Sample

The collected waste samples were subjected for proximate analysis to estimate the fat content, moisture content, protein level and residual ash content present in it. Three set of samples were taken and prepared for analysis as per the standards. The final values were calculated based on the average value of three samples. The overall proximate analysis was carried out based on ASTM standards.

Rendering of Tallow

Post analysis, the rendering effectiveness was determined for various rendering techniques to optimize the most efficient technique for rendering tallow from the subcutaneous wastes. For optimising the effective technique, three set of samples were taken for determining the rendering effectiveness for each techniques and also increase the accuracy of the result. Wet rendering technique was carried out by finely chopping the wastes and boiling it with water attemperature (70-90oC) maintained slightly below the boiling point of water. The tallow was separated from the non-fatty organic compounds and was mixed along with the water. The mixture was centrifuged to separate fat from water and the separated fat was filtered for residues, followed by water washing. The dry rendering of wastes were carried out at 15 bar pressure and 120OC, by heating the finely chopped wastes inside a steam jacketed container, which forces out the fat from the fat-protein matrix and the rendered fat was filtered from any residues followed by water washing.

Pre-Treatment of Tallow

The rendered tallow was heated slightly above $100^{O}C$ to remove any residual moisture content absorbed during rendering or washing process. The dehydrated tallow was degummed by adding 1% of Orthophosporic acid and subjecting it for heating at $60^{O}C$ under continuous stirring for 10minutes followed by prolonged heating without stirring for another 10 minutes.

Transesterification of Tallow

The refined tallow was subjected for alkali based transesterification using ethanol as solvent. Ethanol was chosen because of its renewability, low toxicity with good solvent properties whereas Potassium hydroxide was used as homogenous base catalyst owing to its lesser involvement in saponification reaction in case of presence of higher free fatty acid content. The reaction parameters (influencing parameters and operating parameters) governing the reaction was optimised using statistical method and the ranges of various parameters are tabulated below. The total numbers of experimental runs were determined using equation 1, corresponding to the central composite design. Table 1 consolidates the range of various reaction parameters.

$$n = 2^{K} + 2K + 6 \quad (1)$$

Where n is the number of runs, K is the number of independent variables (K=4)

Table 1: Consolidates the Maximum and Minimum Range of Various Reaction Parameters

S. No	Reaction Parameters	Units	Minimum	Maximum
1	Molar Ratio	-	1:3	1:15
2	Catalyst Concentration	%	1	5
3	Reaction temperature	Deg C (^{O}C)	30	90
4	Reaction time	minutes	30	90

Characterization of Biodiesel

Post reaction, the reaction mixtures were separated under the influence of gravity and the biodiesel was settled on the top with glycerol at its bottom. The separated biodiesel was water washed, dehydrated and was subjected for characterization and quantification of Fatty Acid Esters using FT-IR and Gas Chromatography-Mass Spectrometry. FT-IR spectra briefs about the various vibrational activities of different bonds in the fatty acid ester molecule whereas GC spectra characterises the individual fatty acid ester present in the sample based on their respective retention time and their availability was determined based on its corresponding peak area.

RESULTS AND DISCUSSIONS

Estimation of Fat Content

Based on the proximate analysis, the maximum fat content available in the subcutaneous wastes was determined along with moisture content, protein content and residual ash content and the maximum fat content available in collected samples was found to be 92.84%. The higher fat content in subcutaneous wastes was because of adipocytes, a collection of adipose tissues and areolar connective tissues which has lower concentration of protein and moisture content. Table 2 summarises the fat, water, protein and fat content of subcutaneous wastes.

Table 2: Summarises the Fat, Water, Protein and Fat Content of Subcutaneous Wastes

Fat Source	Sample	Moisture Content,%	Protein Content,%	Fat Content,%	Ash Content,%
Subcutaneous waste	**Johnson et al., 1972**	**4.0**	**1.5**	**94.0**	**0.1**
	Sample 1	3.1	2.15	93.12	1.21
	Sample 2	3.54	2.07	92.6	1.02
	Sample 3	2.94	2.32	92.8	1.1
	Average	**3.19**	**2.18**	**92.84**	**1.11**

Evaluation of Rendering Effectiveness

Generally, fat exist as solid at room temperature but melts beyond 40^{o}C. The optimal fat rendering technique was decided by comparing the rendering effectiveness of drying rendering and wet rendering techniques for these discarded wastes. From table 2, it was evident that hot rendering (120^{O}C, 15 bar) technique was found to be much efficient than wet rendering technique for both the wastes irrespective of its fat content and physical nature. The increase in rendering efficiency for dry rendering technique was because of high pressure inside the container in combination with direct contact of wastes with heat by the means of convection. In case of wet rendering (70^{O}C), the hot water was used as rendering medium and was found to be less effective beyond the boiling point of water. Also, the wet rendering techniques tend to coagulate the wastes and make it tedious to separate it from water-fat mixture. Table 3 summaries the maximum rendering yield and rendering efficiency for wet and dry rendering techniques.

Table 3: Summaries the Maximum Rendering Yield and Rendering Efficiency for Wet and Dry Rendering Techniques

Method	Principle	Source	Sample	Maximum Yield,%	Average Yield,%	Rendering Efficiency,%
Wet Rendering Technique	Temperature gradient difference	Subcutaneous Fat	Sample 1	71.25	73.08	78.72
			Sample 2	74.32		
			Sample 3	73.67		
Dry Rendering Technique	Temperature gradient difference and vacuum extraction	Subcutaneous Fat	Sample 1	86.35	86.27	92.92
			Sample 2	86.57		
			Sample 3	85.89		

Optimization of Transesterification Reaction

Waste beef tallow was converted into biodiesel by means of ethanol based; Potassium Hydroxide (KOH) catalysed transesterification. The optimization of this transesterification was carried out with help of design expert 11.0 using response surface method by considering molar ratio, catalyst concentration, reaction temperature and reaction time as input variables since these variables play a key role in the reaction. Molar ratio signifies the amount of ethyl alcohol required to compensate the amount of triglyceride present in the fat in terms of molar concentration whereas catalyst concentration implies the rate at which the reaction can be enhanced. Reaction temperature ensures the liquid phase of the animal fat and imparts a key role in speeding up the rate of reaction. Even though, catalyst and temperature improves the rate and speed of the reaction, sufficient reaction time must be given for maximum conversion of tallow into biodiesel. The reaction parameters were optimised by maintaining the yield of biodiesel as the output variable. A total of 30 runs, deduced from equation 1, were experimented, based on the different combinations of these variables as mentioned in table 1.

Figure 1 illustrates the surface plot of yield for varying molar ratio and catalyst concentration. It can be noted that yield of biodiesel improved as molar ratio increased from 1:3 to 1:9 since increasing molar ratio favoured stoichiometry of the reaction whereas further increase in molar ratio from 1:9 to 1:15, decreased the biodiesel yield owing to excess availability of ethanol for thorough completion of the reaction. Similarly, it was noted that catalyst concentration favoured the reaction and improvised the yield up to a certain range (2-4% only), beyond which the reaction yielded very less amount of biodiesel irrespective of higher availability of ethanol. The optimum catalyst concentration was found to be 3% which yielded the highest amount of biodiesel (94%). On the contrary, higher concentration of catalyst favoured formation of soaps (potassium salts of fatty acids) irrespective of lower FFA concentration.

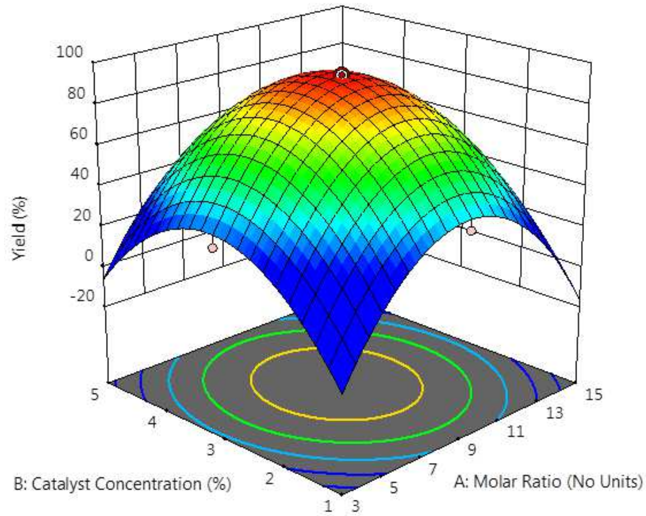

Figure 1: Effect of Molar Ratio and Catalyst Concentration on Biodiesel Yield

Figure 2 illustrates the surface plot of yield for varying molar ratio and reaction temperature and was found to be effective for temperature ranging between 40^{O}C and 70^{O}C. Like catalyst concentration, the yield of biodiesel increased with increase in temperature from 30 to 65^{O}C and reduced gradually as temperature further increased to 90^{O}Cirrespective of higher availability of ethanol. This was because at lower temperature, tallow existed in incomplete liquid phase which doesn't react properly with the solvent whereas at high temperatures ethanol tends to get evaporated thereby reducing their adequate availability for reaction. Highest yield of biodiesel was noted at 65^{O}C which caused the fat to exist in liquid phase as a result of weak Vanderwaal's force of attraction due to continues heating and exhibits higher affinity towards strong nucleophilic ethyl ion [14]. Contrary to expectations, at higher temperature there is a chance of thermal decomposition of tallow which might affect the yield drastically.

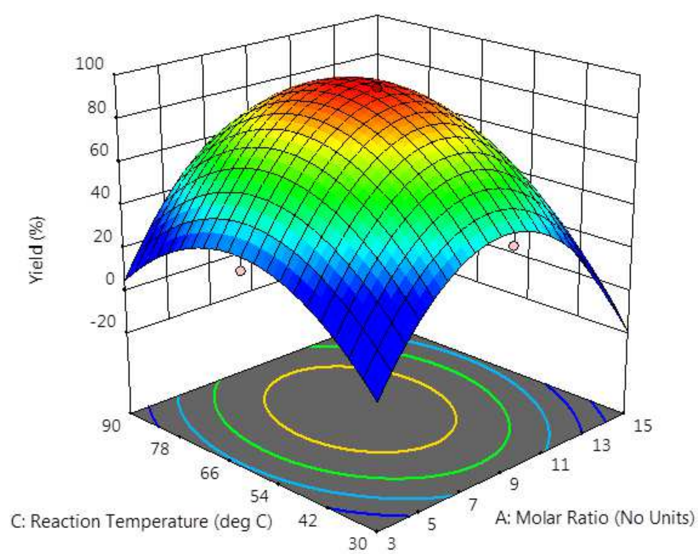

Figure 2: Effect of Molar ratio and Reaction Temperature on Biodiesel Yield

Figure 3 illustrates the surface plot of yield for varying molar ratio and reaction time and was found to be efficient beyond 50 minutes. It was noted that the biodiesel yield was poor for lower reaction time and improper stoichiometric conditions. However, the conversion yield was very effective when the reaction time was maintained between 60-70 minutes and was in good accordance with existing studies and literatures. Thus, it was inferred that average reaction time for transesterification of waste beef fat can be maintained between 60-70 minutes. In addition to that, gradual decrease in the yield was noted as reaction continued further and this time lapse resulted in favouring of reverse reaction upon achieving equilibrium[30].

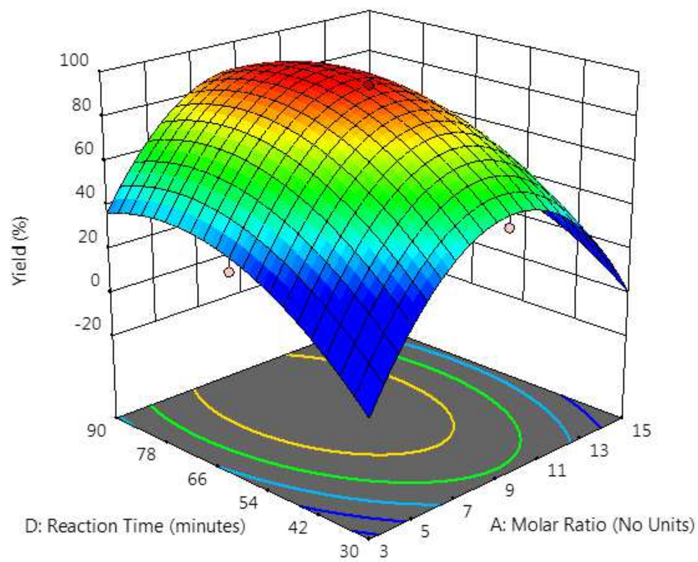

Figure 3: Effect of Molar Ratio and Reaction Time on Biodiesel Yield

Figure 4 illustrates the surface plot of yield for varying reaction temperature and reaction time. It was concluded that maximum yield of biodiesel was noted for reaction temperature of 65^{O}C and reaction time of 70 minutes. It was also inferred that yield reduced gradually with respect to reaction time whereas yield reduced very drastically with respect to reaction temperature. The reduction in yield beyond the optimized reaction time was found to be very less than compared to the yield considered before the reaction time. However, drastic changes in yield was noted at lower temperatures but was less in case of higher temperature and this was because of improper liquid phase at lower temperature. At the same time, higher reaction temperature leads to the volatilization of ethanol thereby reducing the yield.

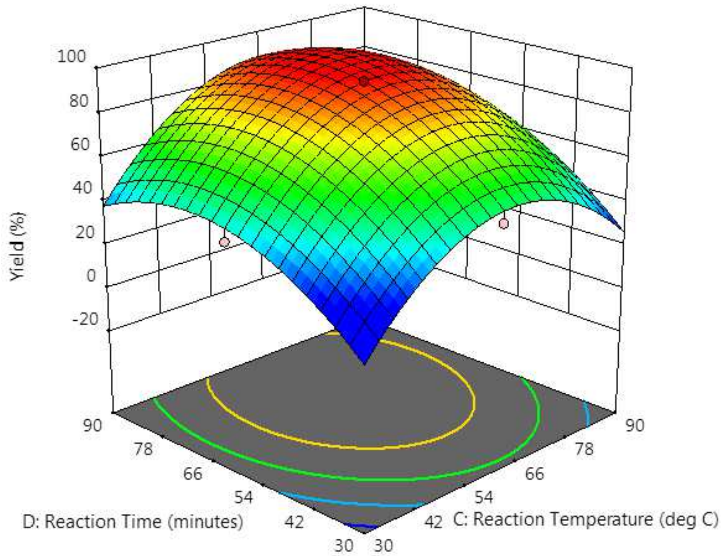

Figure 4: Effect of Reaction Time and Reaction Temperature on Biodiesel Yield

From the experimental runs, most optimized reaction parameters were found to be as follows: (i) tallow to ethanol ratio: 9.5, (ii) catalyst concentration: 3%, (iii) reaction temperature: 65^{O}C and (iv) reaction time: 70 minutes. An empirical equation for determining biodiesel yield was developed by correlating these four reaction parameters and was compared with the actual yield for the corresponding parameters equation 2 denotes the empirical correlation between yield and other reaction parameters.

$$Yield=91.17-6.13A-16.07B-1.01C-0.77D+0.01AB-0.01AC+0.01AD+0.02BC+0.002BD-0.001CD+0.34A^2+2.43B^2+0.01C^2+0.01D^2 \quad (2)$$

Where, A- molar ratio, B- catalyst concentration, C-reaction temperature, D- reaction time

By substituting the optimised parameters in equation 2, the maximum theoretical biodiesel yield was found to be 95.68% whereas maximum actual biodiesel yield was found to be 94.76% which was found to be 1% lesser than the predicted model. The standard deviation between the predicted model and theoretical model was found to be 0.65.

FT-IR Spectra of Tallow Biodiesel

The conversion of tallow into biodiesel was confirmed by the stretching and bending of carbon, hydrogen and oxygen bond at different wavelength using FT-IR. Table 3 summarises the various dominant peaks of the FT-IR spectra of fat biodiesel and tallow. The confirmation of biodiesel is always concluded from the high intensity peaks in the spectra which corresponds to the C=O stretching and O-CH_3 group. The C=O stretching band was noted at 1740 cm^{-1} and signifies the stretching between the carbon and oxygen (C=O bond) bond belonging to the ester functional group present in the biodiesel structure. Similarly, the O-CH_3 band was noted at 1196 cm^{-1} and signifies the alkoxyl stretching between the carbon and oxygen (C-O bond) bond belongs to the ethyl functional group to the carboxylate chain. Figure 5 illustrates the FT-IR spectra of biodiesel and beef fat. Table 4 summarises the various dominant peaks of the FT-IR spectra of fat biodiesel and tallow

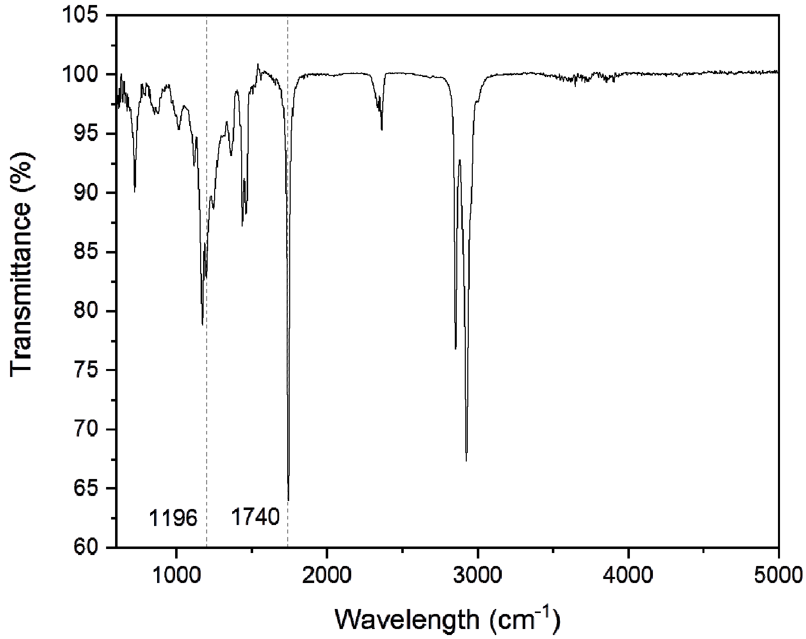

Figure 5: FT-IR Spectra of Waste Tallow and Tallow Biodiesel

Table 4: Summarises the Various Dominant Peaks of the FT-IR Spectra of Fat Biodiesel and Tallow

Wave Number (cm^{-1})	Functional Groups	Bond Activity
1740	C=O Group	Stretching
1196	CH_3-O	Alkoxyl Stretching
2922 & 2852	C-H Stretch	Asymmetrical Stretching
1460	CH_2 Bend	Bending vibrations of the CH_2 and CH_3 aliphatic groups
1433 - 1436	CH_3Asymmetric	Asymmetrical stretching and Rocking vibrations of CH bonds
1168 - 1170	C-CH_2-O Vibration	C-O Stretching

GC Spectra of Tallow Biodiesel

Based on the GC-MS spectra, the dominant fatty acid esters present in the beef tallow biodiesel were characterized and quantified. Ethyl Oleate, Ethyl Palmitate, Ethyl Stearate and Ethyl Myristate were identified as the dominant fatty acids with availability of 40.25%, 21.26%, 17.41% and 3.59% respectively. Table 5 summarises the dominant fatty acid esters present in the beef fat biodiesel.

Table 5: Summarises the Dominant Fatty Acid Esters present in the Tallow Biodiesel

Carbon Number	Fatty Acid Ester	Common Name	Retention Time (min)	Availability %	Molecular Weight (g/mol)
C18:1	9-Octadecenoic Acid Ethyl Ester	Ethyl Oleate	20.687	40.25	310.2
C16:0	Hexadecanoic Acid Ethyl Ester	Ethyl Palmitate	18.75	21.26	284.3
C18:0	Octadecanoic Acid Ethyl Ester	Ethyl Stearate	21.25	17.41	312.3
C14:0	Tetradecanoic Acid Ethyl Ester	Ethyl Myristate	16.75	3.59	256.2

CONCLUSIONS

Thus, the feasibility study of biodiesel production from waste beef subcutaneous fats has been successfully studied and was concluded that possibility of biodiesel production from these wastes were found to be very high. The maximum fat content available in these wastes serves as a viable replacement as low cost feedstock and can be effectively rendered through dry rendering technique. The optimised reaction parameters yielded the highest theoretical biodiesel yield which was 1% higher than that of actual yield. The FT-IR spectra confirmed the conversion of tallow into biodiesel whereas GC spectra characterised the various fatty acid esters present in the beef tallow biodiesel. These results strongly support the viability of biodiesel production from discarded wastes and could pave a way towards cleaner environment through sustainable development.

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